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AD-A228 935

TECHNIQUES FOR INVESTIGATING THIN-FILM INSULATORS  
UNDER HIGH ELECTRIC FIELDS

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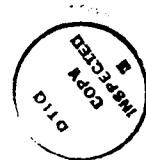
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# ROYAL SIGNALS AND RADAR ESTABLISHMENT

RSRE Memorandum 4401

TITLE: Techniques for investigating thin-film insulators under high electric fields  
AUTHOR: M I J Beale  
DATE: September 1990



## SUMMARY

*are described.*

This memorandum describes techniques developed to investigate thin-film metal-insulator-metal devices operating under high electric fields, and is intended as a practical guide to the experimentalist. Typically, the insulator will be a wide band-gap, optically active material with device applications including displays and electro-optic components. ~~This memorandum contains a battery of approaches, some novel, some not, which provide a strategy for the acquisition of reliable data on this type of sample.~~ Emphasis is placed on techniques to cope with the common problems of non-uniform conduction and of dielectric breakdown. Techniques are described for the measurement, in near real time, of both the electrical characteristics and the optical spectral response. Reference is made to the behaviour of the ZnS:Mn electroluminescent system to illustrate the techniques described. (RH)

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# **Techniques for investigating thin-film insulators under high electric fields**

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# 1. Introduction

This memorandum describes techniques and facilities developed to investigate thin-film insulator devices operating under high electric fields. Its main aim is to provide the experimentalist with a practical working guide to the acquisition of reliable electrical and optical data.

Generally, the devices will be metal-insulator-metal structures with a wide band-gap material as the insulator, Fig. 1. The insulator may be optically active, leading to device applications in the rapidly expanding areas of displays and electro-optic components. Insulators are usually relatively "dirty" in comparison with semi-conductors, however, their expanding applications in electro-optic devices forces our attention on them. The combined problems of awkward materials, the use of thin-films and the application of high electric fields and voltages make their investigation difficult. This memorandum contains ideas and approaches, some novel, some not, which provide a strategy for their investigation.

The term high-field is used here to describe fields in excess of  $10^5 \text{ Vcm}^{-1}$ . This restricts our attention to wide-gap insulating materials; narrow-gap semiconductors and metals will pass sufficient current at these fields to result in immediate destruction by Joule heating. Semi-conductors are therefore not directly addressed in this memorandum; however, some comparisons can be usefully made with this better-understood class of materials. Insulators cannot be fabricated to the same purity and crystalline perfection as semi-conductors especially when in thin-film form. They are less well understood theoretically; particularly the behaviour of the hot electrons generated by the high fields and the characterisation of traps and defects. Many of the experimental approaches used in semi-conductor physics are inapplicable, eg Hall measurements and DLTS and other techniques based on modulation of the depletion layer.

These techniques were originally developed to investigate thin-film ZnS:Mn electroluminescent devices for display applications but many of the approaches described are directly applicable to the investigation of other systems involving high fields [1,2,3]. A particular advantage of the ZnS:Mn system was that the EL is emitted in proportion to the local current density and so acts as a local current flow marker. This showed up the occurrence of a number of laterally non-uniform current transport processes which would otherwise have passed unnoticed. This has widespread implications for the investigation of this type of material. Some results from the ZnS:Mn system are described and the possible extension of this approach to other systems is discussed [1-3].

In dealing with insulators under high fields it is necessary to define what we mean by the term breakdown. In the ZnS:Mn system for example, significant current densities can be passed non-destructively through the "insulator" at fields around  $1 \text{ MVcm}^{-1}$ . However, breakdown with catastrophic thermal run-away can also occur and may be either localised or widespread. Breakdown may also occur at low fields due to imperfections in the films. Investigation of all these aspects of breakdown are considered in this memorandum.

Techniques to cope with the common problem of laterally non-uniform conduction are discussed, particularly when arising from filamentary conduction both destructive and non-destructive. Without suitable techniques it is difficult to obtain reproducible data which is characteristic of the intrinsic behaviour of the films and very difficult to interpret that data correctly. The literature has many examples of thin-film characteristics dominated by a number of discrete leakage paths rather than by the intrinsic properties of the film. Simultaneous areal mapping of the samples with a CCTV system during the electrical characterisation was used for the electroluminescent devices to great benefit, proposals are made for extending this approach to other systems.

The memorandum contains many interlinked ideas and approaches which are briefly stated without being discussed in depth. It is hoped that they will assist in the rapid and reliable characterisation of these difficult materials.

## 2. Sample design

In this section we discuss device design approaches. In general, the design most appropriate for the intended application of the device will be inappropriate for research into its mechanisms of operation; it is the design of samples for the characterisation process that is addressed here.

Of particular importance in thin-films under high-field conditions is the presence of imperfections that result in locally enhanced current density. These may be due to variations in either the material properties or the material thickness. While imperfections can be reduced by good experimental practice, real devices at the research stage have not generally reached this stage and it is essential to be able to characterise imperfect films with some degree of reliability. In the screening and assessment of new materials for electro-optic devices, it is particularly important that the characteristics measured are intrinsic to the new material and are not dominated by defects.

### a) Small area devices

The acquisition of reliable data can be made very much more easily by using small area devices. Only in an extremely well developed process will a  $1\text{cm}^2$  thin-film device be free of pinholes and inclusions. In practice, such defects will probably determine the device breakdown threshold field and dominate conduction below that field. This common-sense point is, however, frequently ignored and cannot be over-emphasised.

For example, a  $100\mu\text{m}$  diameter device ( $10^{-4}\text{ cm}^2$ ) is relatively easy to handle experimentally while giving 4 orders of magnitude greater chance of obtaining a defect free device. Such a device can be fabricated by confining the top electrode to a  $100\mu\text{m}$  diameter on an otherwise large area device. Probing a sample less than  $30\mu\text{m}$  becomes more difficult to fabricate (see 2b) and to probe.

An obvious but significant advantage of a small device area device is that the whole device area can be viewed at high magnification with an optical microscope (eg a  $100\mu\text{m}$  device conveniently fills the field of view of a x50 objective). This is of benefit if any non-uniform behaviour is occurring. We will return to the advantage of imaging the whole sample in section 3.

A further advantage to the use of a grid of small closely spaced devices is that many measurement of different devices on a single substrate can be made and their close proximity should help with reproducibility of measurements and in assessment of the origins of non-reproducible behaviour.

A disadvantage to the use of small area devices is that for a given current density, the current becomes too small to measure. Edge and surface effects may also begin to dominate. The small area may make characterisation more difficult. The optimum trade-off can only be determined for a particular combination of sample and measurement.

The small sample approach is beneficial in the case of problems due to discrete non-uniformities separated by tens of microns or more. However, problems due to non-uniformities with smaller separation or "continuous non-uniformity" such as surface roughness are not helped by this approach, unless perhaps nanometre scale structures are employed which are beyond the scope of this memorandum. Minimisation of such problems is discussed in section 2b however, sometimes, the thin-film device will be so severely blistered or crazed that even a small area grid can not be located between defects. In desperation, small area contacts can be made on such devices by direct contact to the insulator with the contact probe wire. A large series resistor ( $1\text{Gohm}$ ) may help keep local current densities below thermal breakdown levels. The intrinsic breakdown voltage of the film can sometimes be assessed in this way although reproducibility is likely to be rather poor. IV characteristics obtained in this way are even less reliable.

### b) Sample fabrication

Details of sample fabrication are specific to each programme and are outside the scope of this memorandum. However, several general points specific to high-field transport can be profitably made.



In many devices, especially for electro-optic and displays applications, a transparent conducting oxide (TCO) is employed as one electrode. Materials such as tin oxide, indium tin oxide, cadmium tin oxide (or cadmium stannate) and zinc oxide are used. These are all "black art" materials, non-stoichiometric, temporally, chemically and thermally rather unstable. Commercially available TCO coated glass was found to have handling and cleaning induced surface scratches. Thin-film deposition onto this material may result in current density non-uniformity reflecting the surface scratches, these were clearly visible on my own ZnS:Mn EL devices. If the TCO can be fabricated within your laboratory with thin-film deposition following immediately from TCO deposition then this should ameliorate the surface scratch problem.

Possible deposition techniques for the insulator include sputtering, evaporation and chemical vapour deposition. While the physicist may incline towards the cleanest approach possible, perhaps MBE, this may not always be the best approach for several reasons. Traps may dominate the behaviour of the samples and may be thermodynamic in origin and not due to contamination. The highest levels of cleanliness are not then essential as would be the case in semi-conductor research. Traps may also be beneficial or even crucial to the conduction process. In this type of sample, traps should therefore not necessarily be regarded as detrimental to device operation.

In elucidation of the physical mechanism it may be highly beneficial to have several different fabrication techniques to allow comparisons to be made.

Surface roughness effects arising from orientation dependent grain growth rates in polycrystalline films and from film stresses during either growth or annealing are more difficult to control. The growth of thinner films may be beneficial but the topic of thin-film growth is beyond the scope of this memorandum. Similarly, crazing of the thin-film induced by stresses and possibly by exposure to the ambient can make film essentially un-characterisable. Again thinner films may be beneficial, and avoidance of atmospheric and moisture exposure. Blisters in the thin-film or in the top metallisation may also be problematic.

After thin-film deposition, electrical contact to the top of the film is required. The evaporation of this top contact through a TEM grid as a mask has several attractive features. It is simple, rapid and relatively non-damaging to the sample. In contrast, photolithograph involves more complex processing and has the risk that the wet chemical processing may significantly affect the film's properties. Both sputter deposition and the use of dry etching for pattern definition will cause structural damage to the near surface regions of the thin-film and are best avoided for film characterisation, at least in the early stages of the programme.

Deposition of the top contact can be conveniently achieved by evaporation of aluminium through a transmission electron microscope grid. Standard grids are 3mm diameter thin metal films with an etched pattern of holes with sizes in the range 30 to 300µm. The smallest size that can be used is determined by the sizes available, by the resolution limits imposed by evaporation through a mask and by the problems of making electrical contacts to the grid..

### **c) Self-healing contacts**

It is highly beneficial for thin-film characterisation to employ "self-healing breakdown" top contacts even if they are not practical for the final device application. In the event of a breakdown, there is a localised run-away rise in current density with consequent thermal destruction of the film. This may occur on nano-second timescales and with temperature well in excess of 1000°C. However, if the top contact is a thin, readily vaporised layer, then the local temperature rise can vaporise the nearby top contact to leave the damaged device area electrically isolated. In the ideal case, device operation can then continue, modified only by a small reduction in its total area. In real situations, however, some residual conduction may remain in the heat affected zone around the breakdown. The use of continuous optical and electrical monitoring is therefore recommended.

If a thick and/or refractory top contact is employed, such as a 1µm layer of Cr, then self-healing breakdowns may not occur and a breakdown event may destroy the entire sample. Alternatively, it can happen that local destruction of the insulator occurs within the film with no external sign other than an increase in the conductivity of the device. Such cases can occur when one electrode is of relatively high

resistivity, such that the spreading resistance limits the peak current density to a level that damages, but does not vaporise, the sample.

In addition to the experimental advantage of being able to continue measurement on a device after a catastrophic breakdown event, the use of a self-healing design allows a statistical picture of the breakdown events themselves to be obtained from a single sample.

#### **d) Electrical advantages of small samples**

Electrical measurements are discussed in more detail in section 4. Here we outline some advantages of small devices for electrical characterisation.

Large area thin-film devices will have relatively large capacitance which generally gives rise to a large RC time-constant with consequently restricted frequency range over which data can be gathered. Small devices can therefore have significant advantages for electrical measurements. The time-constant arises from the geometric capacitance of the device in combination with the series resistor which is a part of the external drive circuit necessary to control power surges during a breakdown event, this is discussed in more detail in section 4a. The minimum usable value of this series resistor is independent of the device size and so the RC time-constant scales with device area. For example, the time-constant with a 100 $\mu\text{m}$  device 1 $\mu\text{m}$  thick and the minimum 10k $\Omega$  series resistor is in the region of 1 $\mu\text{s}$ ; with a 1 $\text{cm}^2$  this rises to 10msec.

A further advantage with the use of small samples arises from the need for a series protection resistor of at least 10k $\Omega$  but which is independent of the sample area. The voltage drop across this resistor (IR) scales with device area for a given current density. Even a small IR drop can be problematic; if constant voltage drive (see below) is used then this drop acts as a negative feedback element which can complicate and confuse the interpretation of the data. A rise in total current through the device results in a reduction of the voltage across it, this rise may result from a local increase in current density which then causes the field across the rest of the device to be reduced. For example, a current density of 1mA $\text{cm}^{-2}$  in a 100 $\mu\text{m}$  diameter device results in a voltage drop of 1mV across a 10k $\Omega$  resistor. While this may be acceptable, a 1 $\text{cm}^2$  device gives a drop of 10V. The situation becomes still more serious with increasing current density in the device.

Yet another advantage of small devices is that in the event of a breakdown event, the amount of energy deposited during the breakdown is approximately  $1/2 CV^2$ , see section 2c. Again this scales with device area and in practice the size of the crater left after a self healing breakdown has occurred does increase in proportion to the device area via this effect.

#### **e) Special sample designs**

The preparation of a thickness stepped sample can be very beneficial to understanding the properties of the thin-films, Fig 2. For example, in a study of high-field transport mechanism, we can distinguish between interface controlled and bulk controlled transport by plotting the voltage for a specific current against sample thickness. The linearity of the characteristics and their intercepts with the axes allow deconvolution of bulk and surface effects. If, within a single deposition run, a sample is prepared with stepped thickness, then the data can be gathered elegantly and rapidly without the worry of run to run thin-film reproducibility. Thickness dependent morphology changes are still a concern however.

A stepped thickness sample can be prepared during growth by the step-wise withdrawal of a shutter placed between the sample and the sputtering target, Fig 2. A stepped sample is more useful than a ramped sample to avoid thickness variation across a sample and to ease the accurate measurement of sample thickness using a surface profilometer.

Deposition of a transparent conducting oxide as the top contact may be attractive, particularly for light emitting devices. Problems with short-circuits induced by the relatively aggressive sputtering process may be significant. The general requirement for deposition at an elevated temperature or a post deposition anneal may also cause problems.

## **f) Light emitting samples**

Electroluminescence in the ZnS:Mn system proved an extremely valuable tool for understanding the current transport processes in those devices. Over a wide range of current densities, the EL brightness is directly proportional to the current density; at high current densities the EL brightness becomes sub-linear. Spatial mapping of the local current density is therefore visualised directly by the imaging system. Crucial observations about current filamentation were made that would have passed unobserved without the EL to make them visible [1]. Locally high current density due to film imperfections can be identified which is very important in ensuring that electrical characteristics are characteristic of the whole device and not just a few discrete leakage paths. Information about the hot electron distribution within the depth of the film can also be probed by insertion of a stripe of Mn in a Mn free sample.

Further advantages of monitoring the EL emission during electrical measurement makes use of the EL brightness being directly proportional to current. If the current-brightness characteristics do not intercept the origin then this is indicative of a "leakage" current not giving rise to EL, generally due to discrete leakage paths. Absence of such pathological behaviour is a valuable indication that uniform conduction is occurring and that the electrical data are trustworthy.

It may also be possible to extend the benefits of this approach to devices not deliberately fabricated to emit EL. In such systems, the high-field conditions may still give rise to EL at the band-gap energy or below. These emissions may be worth looking for as they would provide an extremely valuable spatial mapping tool for current density. It is noteworthy that in the case of Mn doping of ZnS to approximately 0.5% Mn bright and efficient EL devices can be fabricated and yet the Mn has little or no effect on the current transport in the ZnS. It may be possible to dope the insulator in your own MIM structures to provide EL emission for spatial mapping without affecting the parameters of importance.

An alternative and novel approach would be to use a phosphor to down-convert any high energy band-gap emission into the visible. The phosphor could be a film deposited adjacent to the thin-film or a layer deposited on top of a very thin semi-transparent gold electrode. Note that transparent conducting oxides start to absorb strongly in the UV energies associated with band-gap emission from these wide-gap materials under investigations.

## **g) Transverse field configuration**

In the devices so far described, the field has been applied across the thickness of the film. This necessitates deposition on a conducting substrate which is not always possible or appropriate. Here we consider the use of an electrode structure deposited on only one surface of the film with a structure enabling a transverse field to be applied to the sample. The electrodes may be deposited either after or before the growth of the dielectric film.

An inter-digitated structure may be employed such that the fraction of the device under the high field is increased to facilitate optical measurement. Problems associated with the use of transverse field devices include the need for either very large voltages or very small lithography geometries if MV/cm fields are required. These fields lead to air breakdown problems and it may be necessary to operate the device in vacuum, under silicone oil or with a further dielectric layer encapsulating the device. A serious concern for interpreting the electrical characteristics is the relative importance of bulk and surface (interface) conduction paths. Electrical data obtained on transverse devices may not relate to those obtained on through-thickness devices. However, optical measurements can still be conveniently made on transverse devices as the electric field distribution is relatively easily determined.

### 3. Equipment design

Here we outline the hardware developed to characterise thin-film high-field devices including the cryostat, probing system and CCTV system. The electrical and optical characterisation techniques are described in sections 4 and 5.

In general, thin-films are likely to be sensitive to the ambient atmosphere and measurement should be made in a controlled environment. A vacuum cryostat is ideal as it also allows physical mechanisms to be probed via their temperature dependence. A means of probing the sample is required and CCTV facilities are needed to monitor the sample visually and to record the results for subsequent examination.

#### a) Cryostat design

The XYZ probing of the sample (see section 3b) and the need to optically examine the samples during electrical characterisation are likely to necessitate a specially constructed cryostat. Mine was based on Polaron's LN<sub>2</sub> cryostat using their temperature control system LN<sub>2</sub> pump and electrical heater and sample mounting. An 18" diameter HV flange allowed sufficient space for the XYZ motors. A key requirement is a thin window with minimal optical path to the sample. Even with ultra-long working distance microscope objectives care is needed to keep the working distance down, Fig 3.

In the case of transparent samples, illumination from below the device is highly desirable to help locate the probe and to identify breakdown events, pinholes, Al oxidation effects etc. This requires either a hole in the sample mounting plate below the active device or, more convenient for probe manipulation, a sample that hangs over the edge of the sample mounting plate. Thermal contact is then more problematic and has been assisted by using very thin mica for electrical isolation and a stainless steel spring loaded tape to force the sample into good thermal contact. Vacuum grease can also be used to improve thermal contact but this tends to creep badly at elevated temperatures. Auxiliary thermocouples and out of contact thermometry can also be employed to ensure accurate thermometry.

It is desirable to be able to illuminate the sample while it is in the cryostat and being imaged by the CCTV microscope system. Photo-conductivity measurements are a useful analytical tool in trap investigation and the wavelength dependence of the optical properties is crucial to many electro-optic devices. An externally mounted mirror and lens system has been used to direct light from a laser or monochromator onto the sample active area.

It is essential to protect the CCTV system from over exposure to light, particularly if lasers are being used. In the case of laser illumination, a thin-film interference filter can conveniently be slotted into the centre of the microscope optical to protect SIT, PM tube and eyes from exposure without seriously reducing the transmission of other wavelengths. In the case of relatively broad-band or variable wavelength illumination, polarising filters on the monochromator output and in the microscope optics can be employed.

#### b) XYZ probe

The recommended approach to electrically contact the sample is to use a fine wire and an XYZ motorized manipulator. Gold bonding wire of approximately 50µm diameter, doped with 3% Ga provides a suitably springy and corrosion free contact; pure gold is insufficiently springy. The wire may conveniently be supported by passing it through a hypodermic needle which can then be fixed to a motor driven XYZ control with travel of several cm in each direction (eg Ealing Optical Co).

The characterisation of a large number of devices is assisted by the combination of a grid of closely spaced samples and the use of the XYZ contacting system. It is a major practical advantage to be able to measure many closely spaced devices on a single substrate to assess reproducibility of both the devices and the data acquisition. With the XYZ probing approach, multiple repositioning of the probe allows an assessment to be made of the importance of device damage by the probe contact. The use of a motor driven

XYZ probe has the advantage that probing is possible in a cryostat or other controlled environment provided vacuum compatibility of the motor drives is considered. It is a major practical advantage in this high-field work to be able to probe many samples without having to break vacuum and return to room temperature each time.

#### b) Alternative probing methods

An alternative to the XYZ probing is wire-bonding (thermal, ultra-sonic, pressure bonded etc). Wire bonding is much less convenient if many samples are to be measured or if the samples are in close proximity to one another. Furthermore, there is the worry of device damage by the contact forming process that is best eliminated in the early stages of the investigation.

Contact could also be made using a mercury probe, however, this is not feasible under vacuum conditions. Contact can also be made by other liquids including water and ethanol. These provide transparent but rather uncontrollable contacts.

#### c) CCTV microscopy

A silicon intensified target (SIT) camera was used to image the samples at TV rates. This system has an integral image intensifier and offers a good sensitivity ( $10^{-4}$  lux), freedom from artifacts and cost compromise. Due to the high magnifications required to study filamentary flow and other laterally non-uniform behaviour, a very light-sensitive camera is required. A clear video-rate image is then obtained under conditions that the dark adapted eye looking down the microscope can see nothing. Greater light sensitivities are obtainable than with the SIT but at much greater costs (eg see Hamamatsu systems).

Simultaneous CCTV imaging, imaging by eye (in colour and with better resolution and dynamic range) and quantification of total light emission by photomultiplier is achieved with the following configuration. An optical microscope with binocular viewers and an output for photo-microscopy is used. The SIT camera is attached to the microscopy output and the PM tube is fixed in place of one of the binocular eyepieces. An eye can be applied to the other eyepiece which should otherwise be blanked off to prevent ingress of light. The XY motion of the microscope over the sample fixed in location in the cryostat is best achieved by the fabrication of a large XY slide system to which the microscope is fixed. Construction must be massive to minimise vibrations and to carry the weight of the microscope and SIT camera cantilevered over the cryostat system. Other requirements on the microscope include ultra-long working-distance objectives (eg Olympus microscopes).

The use of a VCR with good freeze frame facilities is very useful in the study of transient or kinetic effects. Breakdown studies require the ability to re-examine the event, to view it in slow-motion and with time reversed. A major problem is that many breakdown events are on timescale orders of magnitude shorter than the CCTV 40 msec frame rate (20 msec field rate). Insertion of a strobe shutter (eg Bentham Instruments) phase locked to the TV frame has allowed one or more sub-millisecond exposures per frame. However, with very short exposures, too little light intensity is available except for exceptionally bright events such as a destructive localised breakdown.

It was central to the characterisation of the ZnS:Mn EL samples that they be visually examined during the electrical measurement process. This proved absolutely essential to making progress in understanding the electroluminescent devices and a number of laterally non-uniform and time-dependent behaviours were discovered which had previously gone un-noticed. In the case of EL devices, the light emission acts as a marker for current flow. The local EL emission is directly proportional to the local current density over a wide range of operating condition and so can be used to map spatially the local current density with an attainable resolution of approximately 1 $\mu$ m. The extension of this behaviour to other systems was discussed in section 2f.

#### **d) Photometry**

A PM tube is fixed in place of one of the binocular eyepieces on the microscope to allow simultaneous CCTV monitoring, monitoring by eye and quantification of total light output by PM tube and current to voltage amplifier. A high sensitivity tube operated at its optimum signal to noise ratio is generally required.

#### **e) Birefringence and spectral analysis**

The optical birefringent properties of a small area sample can conveniently be probed using the CCTV microscope system already described. The sample is illuminated with polarised light using the microscopes normal reflected light illumination system in conjunction with a polarising filter. The illuminated area and its location are then automatically appropriate for the particular objective lens in use. The analyser is then positioned in the optical path such that only the reflected light from the sample passes through it before being imaged. Rotation of polarised and analyser can be performed to look for changes in the extinction conditions as a function of applied electric field. The use of polarised sub-stage illumination can similarly be used to determine the transmission characteristics. Both normal and transverse field devices can be assessed in this way.

Spectral analysis facilities can be relatively easily incorporated into the equipment using a Monolight Instruments Ltd. Scanning Monochromator. This system employs a mechanically driven grating system and a photomultiplier to provide ten spectral scans per second. These may be conveniently captured on a digital storage oscilloscope for signal processing and recording on a computer system. The light from the sample can conveniently be introduced into the spectrum analyser from one of the microscope eye-pieces. This is facilitated if the microscope has an angle adjustable eyepiece which can be brought to the horizontal position to align with the monochromator optics. Simultaneous spectral analysis with one eyepiece, PM light measurement with the other eyepiece and CCTV monitoring with the camera port is then possible.

The light to be analysed may either be generated within the sample or by an external source of illumination. In the latter case, the standard microscope illumination system is ideal as the area illuminated is then automatically of the appropriate size for the objective lens being used and its location is visible on the CCTV system.

It is desirable to protect the eye, the PM tube and the SIT camera from damage by the illumination by locating an appropriate filter within the optical system. In the case of laser illumination this filter becomes essential. A convenient source of suitable filter material is the lens from laser goggles.

### **4. Electrical measurements**

This section discussed equipment, circuits and techniques for electrical characterisation. It is emphasised again that simultaneous visual monitoring, and perhaps optical characterisation are valuable aids to ensuring valid data are gathered.

#### **a) Series resistor consideration**

It is advisable to include a series resistor in the device drive circuit in order to limit the extent of the device destruction in the event of breakdown. The resistor has two possible and independent roles. It may influence the initiation of a breakdown event; however, this is unlikely. The main role of the resistor is to affect the development of a breakdown event.

If a high field is applied to the device then it is usually essential to have a resistor of approximately 10kohm in series with the sample, this is independent of the sample size. This value is sufficient to isolate the device from the power supply during the time-scale of the breakdown event so that a self-healing breakdown rather than total sample destruction occurs. If the high field is only present for short pulses (micro-second or less) then the series resistor may not be necessary as the total energy flow from the power supply is then limited by the duration of the pulse itself.

Unfortunately, this resistor, in association with the intrinsic device capacitance, gives rise to an RC time-constant giving an upper limit to AC measurement frequencies and a lower limit to the time-scale that can be probed in transient analysis. Capacitance can be minimised by the use of small area devices, for example, the time-constant with a 100um device 1um thick and a 10kohm series resistor is in the region of 1usec; with a 1cm<sup>2</sup> this rises to 10msec.

A further disadvantage of the series resistor is the voltage drop occurring across it (IR) which also scales with device area. If constant voltage drive (see below) is used then this drop acts as a negative feedback element which can complicate and confuse the interpretation of the data. A moderately high current density of 1Acm<sup>-2</sup> in a 100um device results in a voltage drop of 1V across a 10kohm resistor, this may be quite significant as the high-field IV characteristics generally have very steep slopes. With a 1cm<sup>2</sup> device the drop becomes an unacceptable 10kV.

#### b) DC equipment

High impedance devices require electrometer grade voltage sensing with input impedances of 10<sup>14</sup> ohms; the use of lower grade instruments can give totally erroneous results. High capacitive loading may also result in catastrophic damage in the event of a breakdown event by virtue of the large storage energy  $\frac{1}{2}CV^2$  as previously discussed.

High fields can result in high voltage generation and measurement requirements necessitating special equipment and great care. In the case of electro-optic devices where some films are necessarily 5 to 10um thick the voltage requirement can become horrifying! Keithley instruments 617 electrometers go to 105V, their 237 supply and measuring system goes to 1100V.

A further effect is that in the event of a breakdown event, the amount of energy deposited during the breakdown is approximately  $\frac{1}{2}CV^2$ . Again this scales with device area and in practice the size of the crater left after a self healing breakdown has occurred does increase in proportion to the device area via this effect.

It is highly desirable to make measurements under both drive polarities. These help to elucidate many aspect of film behaviour including the roles of contact effects in conduction and the mechanism of any electro-optic effects. As the electrometer systems do not have a floating input and the requirement to switch polarities while measuring both sample voltage and current necessitated the development of a simple switching box, Fig. 4.

Both constant voltage and constant current power supplies can be employed. Almost all of my work was constant voltage supplies for the reasons to be outlined. The Keithley constant current supply K220 has a settling time of milliseconds, any constant current instrument must have a settling time associated with the feedback loop required to control the current. In practice, this settling time was found to result in significantly more frequent device breakdown, presumably due to over-volting during the settling period. Furthermore, the breakdowns were less likely to self-heal successfully. This was a particularly serious problem in practice and made constant current drive impractical for many experiments. This may be because the sudden drop in voltage on the device after a breakdown takes much longer to recover with constant current drive thus effectively reducing the possibility of subsequent breakdown at the same site which are often observed to occur before the site is fully self healed. The mechanisms proposed here are speculative at this stage, however, the practical disadvantages of the more convenient, constant current drive were repeatedly observed.

A more fundamental reason for using constant voltage drive lies in the MIM sample design. The two conducting electrodes of the MIM structure ensure that within the device there is a laterally uniform voltage across it regardless of external circuit considerations such as constant voltage or constant current drive. If any lateral non-uniformity of conduction occurs then it becomes impossible to relate the measured total device current to locally varying current density, whereas the measured voltage across the device is still a unique and position independent parameter. The MIM structure is therefore essential a constant field structure in the event of non-uniform processes occurring and constant voltage drive is more natural as well as practically more successful.

In the event of wishing to continue device operation and characterisation after only partially successful self healing breakdown events then constant voltage drive becomes essential as progressively increasing leakage current would otherwise starve the remaining device area of current.

The use of IEEE computer control from an IBM AT type computer running the language ASYST proved highly beneficial to the simultaneous control and logging of current, voltage, brightness, temperature and time data. However, this is outside the scope of this memorandum.

#### **c) Pulse equipment**

In addition to quasi-static DC measurements, electrical characterisation requires time-domain or pulse measurements. The equipment required for thin-film high-voltage investigations is briefly described.

A waveform synthesiser was a convenient and versatile source of the drive waveform (eg a Wavetec 2020). The high voltages often required for these devices requires a special pulse amplifier circuit to be used. This is based on the relatively fast, high voltage Burr Brown op-amp 3584JM. Fig. 6.

A high voltage buffer amp. based on the same device was also required to prevent the resistive and capacitive loading of the high impedance devices by the oscilloscope, Fig. 5. A four channel digital storage 'scope is desirable to monitor voltage, current and brightness waveforms (eg LeCroy 9400; IEEE is also desirable for signal processing).

A PM tube with a low noise fast amplifier is required. As the noise-frequency response trade-off is critical in pulse measurements, it is best to have start with a wide-bandwidth low-noise current amplifier with a separate RC filter to optimise its band-width for the signal under examination. Considerable further signal to noise enhancement can be had by signal averaging with the storage scope. In practice, both approaches had to be used with the ZnS:Mn devices.

#### **d) AC Impedance equipment**

In addition to DC measurements and pulse measurement, electrical characterisation requires frequency-domain or impedance measurements. The equipment is briefly described.

A solartron 1260 impedance analyser was employed. The internal programmable bias circuitry is of little help for this type of sample as the voltage source is limited to 6V with DC coupling and to  $\pm 40$  V with AC coupling. For this application, use the instrument with DC coupling throughout as internal blocking capacitors produce spurious results on my samples that were not readily identified. Use high grade polycarbonate capacitors to block the externally applied DC bias from the AC voltage source and from the voltage sensing input. The current input must be left AC coupled but will be unharmed as its impedance to ground is low (approximately 100 ohm) and thus harmful high voltage will not be generated, see Fig. 8

Interpretation of the impedance results require care and thought. So-called negative capacitance effect may be observed due to time dependent behaviour of MIM structures [2]. Ensure that sufficient currents are registered in both Real and Imaginary components or errors will be large. Ensure that instrumental interpretation of the meaning of the current components is based on correct circuit identification or spurious results will be generated.



## 5. Optical characterisation

Some optical characterisation procedures are now briefly outlined. The original project from which this memorandum grew was aimed at understanding electro-luminescent ZnS:Mn devices for display applications. The optical characterisation was primarily concerned with the measurement and spatial mapping of relatively bright electroluminescent output of yellow colour (588nm). Extension to this approach to other MIM systems was outlined in 2f. We briefly outline these and some other types of optical characterisation that can be integrated into the system.

### a) Electroluminescence

Electroluminescence in the ZnS:Mn system from which this memorandum arises, proved an extremely valuable tool for understanding the current transport processes in those devices. Simultaneous DC electrical and EL monitoring can be performed using a Keithley 617 electrometer to measure the current generated in a photomultiplier.

Measurement of time-dependent EL response requires the use of a low noise fast current amplifier as discussed in section 4c.

### b) Photoconductivity

The effects of illumination on the electrical characteristics of a thin-film device can provide useful information on conduction mechanisms especially the role of traps. HeNe (red 633nm 1.96eV), HeCd (blue 422 nm 2.94eV) and QI and Xe based monochromator light can be used to examine photoconductivity and electro-optic effects. A tunable source of illumination that can go up to the band-gap energy is useful for trap characterisation.

Within the cryostat, illumination from both above the device and below is desirable to assist in probe manipulation and in the identification of self-healing breakdowns.

### c) Refractive index and electro-optic constant changes

The spectral transmission or reflection response of filter structures and other electro-optic components requires the use of a system such as that described in section 3 e). The illuminating radiation should be absorbed by a suitable filter as previously described. The field dependence of electroluminescent emission, transmitted light or reflected light can be determined in near real time using this approach. The area to be measured can be selected according to the electrode geometry, the objective lens and the use of apertures. Both normal and transverse devices can be assessed. The CCTV system can provide spatial information about the electro-optic effects.

## 6. Some typical device behaviour

A brief outline of various aspects of the behaviour of a typical ZnS:Mn thin-film EL sample is now presented. It gives valuable clues as to what may occur in other samples. The advantages of the EL emission as a marker for the lateral mapping of current density will be made very apparent. In systems with no such marker, some aspect of this behaviour will almost certainly occur and the unwary experimentalist may be seriously misled.

### a) Very low field conduction

As the voltage is applied for the first time to a virgin device, and is slowly ramped up from zero,

significant conduction at very low fields may be observed. This is particularly likely with a large area sample but may be absent in small area samples. In practice, this conduction may make it impossible to apply a high voltage (high-field) to the device with the results that, for example, EL or electro-optic effects, may not be excited. This behaviour is characteristic of one or more discrete high conductivity paths arising from film defects, pinholes etc. Their characteristics may be ohmic but are not necessarily so. Sometimes, the high conductivity paths will burn out to leave a functioning device. This process can be encouraged by either transient reduction of the series resistor to allow the passage of large currents. This may clear the leakage paths in a self-healing manner, however, this procedure can not be relied upon and the use of small area samples to reduce the probability of such defects is preferable.

#### **b) Low field conduction in filaments**

If a device is measured that is free from very low field conduction, then as the voltage is increased it may show EL emission from several discrete points at an applied field too low to excite uniform conduction and EL emission from the rest of the device. This behaviour is characteristic of a device with thin regions of insulator perhaps due to dust in the film or to scratches or dust on the substrate layer. This behaviour is very common in the ZnS:Mn devices where it is clearly visible by the EL emission. This filamentary conduction may well occur in other thin-film devices but passes unnoticed without the EL marker. These conduction paths are of high resistance in low field conditions unlike the short-circuit like defects previously described. A high field can be sustained in the insulator in these regions, as demonstrated by the occurrence of the EL, but occurs at lower applied voltage than in the remaining device. The effect of such conduction paths can be to distort the IV characteristics at low fields by giving apparently enhanced low field conduction. Many examples in the literature are dominated by this effect.

This type of film defect can often be burned out by a self-healing breakdown event. With a virgin device, it is beneficial to ramp up the voltage slowly to encourage self healing breakdown at these weak points. The change in current as a result of such a breakdown gives an idea of the current through that defect region and also of the effectiveness of the self healing. If the top contact is not designed for self-healing, then a permanent conduction path may be established. In this case, the current through the region may be limited by the spreading resistance of one of the contact electrodes.

#### **c) Polarity dependence**

Detailed discussion of polarity dependent effects is beyond the scope of this memorandum but measurement in both polarities is very strongly recommended for the elucidation of transport mechanisms. A valuable set of clues can be obtained from the polarity dependence of the IV characteristics. Both the probability of breakdown events occurring and also the probability of their being self-healing can be polarity dependent. The appearance of the breakdown craters also tends to be polarity dependent, perhaps giving clues as to the mechanisms of their formation.

#### **d) Interfacial contamination effects**

Again the value of EL devices for mapping current density was demonstrated in the appearance of "contrast reversal" effects. On some devices, the current flow was patchy, Fig 9, this we ascribe to contamination by vacuum oil of the insulator-top contact interface. On reversing the drive polarity, the high and low current regions were interchanged giving reversal of the contrast. The presence of a patchy semi-insulating layer at an interface can inhibit or enhance current density relative to an uncontaminated region. Unnoticed, such behaviour would give erroneous IV characteristics. The effect of the contamination layer indicates that the use of a contact layer that is, at low fields, an insulator, can provide a more efficient carrier injector than a deposited metal film.

#### **e) Intrinsic breakdown**

Breakdown due to film imperfection of various types has been discussed. There is also evidence for breakdown that is not directly related to film perfection. Such intrinsic breakdown may arise from

negative differential resistance behaviour characteristic of the material, from charge trapping effects during the passage of current or from processes such as avalanche multiplication. It is beyond the scope of this memorandum to discuss the mechanisms involved, however, it should be remembered that the achievement of the perfect thin-film may NOT result in a device free of breakdown events under high-field conditions.

#### **f) Current filamentation**

It is self-evident that the breakdown events discussed above are examples of current filamentation arising from either film defects or from material characteristics. Although the origins of these may be athermal, the destructive mechanism is localised Joule heating to temperatures of many 1000°C. However, in the ZnS:Mn sample I have also observed current filamentation that is non-destructive. Sub-micron diameter filaments turn on, last for milliseconds and then self extinguish non-destructively and may combine to form wave-like patterns [1], Fig 11. Although this behaviour may not be wide-spread in MIM devices, it is advisable to be aware of its possibility. Continuous CCTV monitoring of an EL signal is clearly advantageous.

#### **g) Blistering during operation**

The formation of blisters in a device is sometimes observed during operation and should be monitored. This may occur due to gas evolution by an electro-chemical reaction within the films, trapped water may be involved. Blistering may be strongly drive polarity dependent.

#### **h) Partial self-healing breakdowns**

Self-healing breakdowns may leave a residual weakly conducting path behind them, ie self-healing may only be partial. It is essential to know if this is occurring or the current-voltage characteristics measured are those of discrete residual leakage paths and not the uniform part of the device. This is particularly true of relatively low field measurement where the ratio of the total current in these leakage paths to the total current through the uniform part of the device can be very high. At higher field, the uniform conduction may again dominate. This is a major practical problem which has invalidated many examples of data in the literature. Some examples of self-healing craters after localised dielectric breakdown are shown in Fig. 10.

## **7. Conclusions.**

The reader has been alerted to many of the problems which make characterisation of thin-film high-field samples difficult. Reference has been made to the ZnS:Mn system which acts as a model system, in that its electroluminescent emission acts as a spatial marker for the current flow through the film. The results from this system alert us to the many types of non-uniform current flow that can occur. If the experimentalist is unaware of this behaviour then erroneous conclusions will be drawn from the data. The use of a electroluminescent film is strongly encouraged in the work of others; however, where this is impractical, then to be forewarned is to be forearmed.

Characterisation of thin-film, high-field materials has a long and rather undistinguished history due in part to the problems specific to these materials and to the MIM high-field device structure. Insulators are generally dirty and ill understood in comparison with semi-conductors. However, the rapidly increasing demands for large area displays and for electro-optic components is creating greater interest in thin-film materials under high-field conditions. I hope that the techniques and approaches described will assist future investigations of this interesting class of materials.

## Acknowledgements

I am very grateful to John Kirton who initiated the RSRE programme on ZnS:Mn out of which the reported work evolved and with whom I have had many helpful discussions. Also to Mike Slater and John Evans for deposition equipment modifications, sample design and sample fabrication. Thanks also to vacation students Peter Mackay, Mark Welsh, Mike Chapman and Joe Lynass for their contributions to equipment development. I am also grateful to Charles Smith for the high voltage amplifier circuit in Fig 6. Thank you also to my wife for proof reading.

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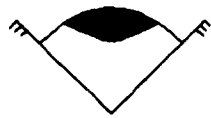
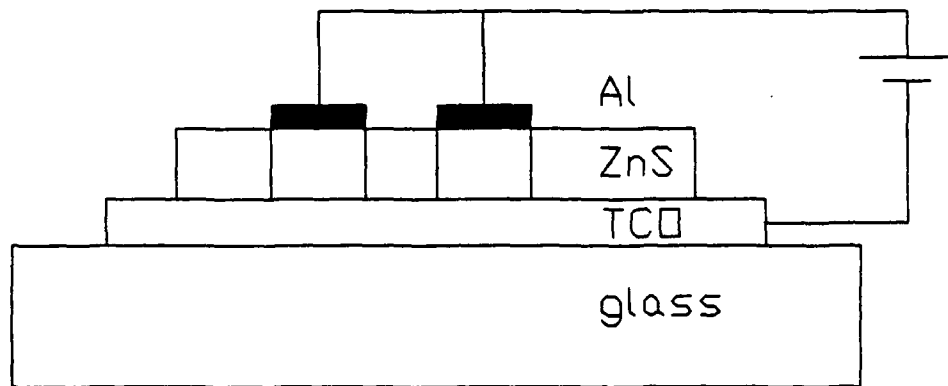
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## **Figures**

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- 3      Cryostat design
- 4      DC switching circuit
- 5      Buffer amplifier (high voltage)
- 6      Pulse amplifier (high voltage)
- 7      Constant current supply
- 8      Impedance analyser circuit
- 9      Interfacial contamination effects
- 10     Self-healing breakdown craters
- 11     Kinetic electroluminescence

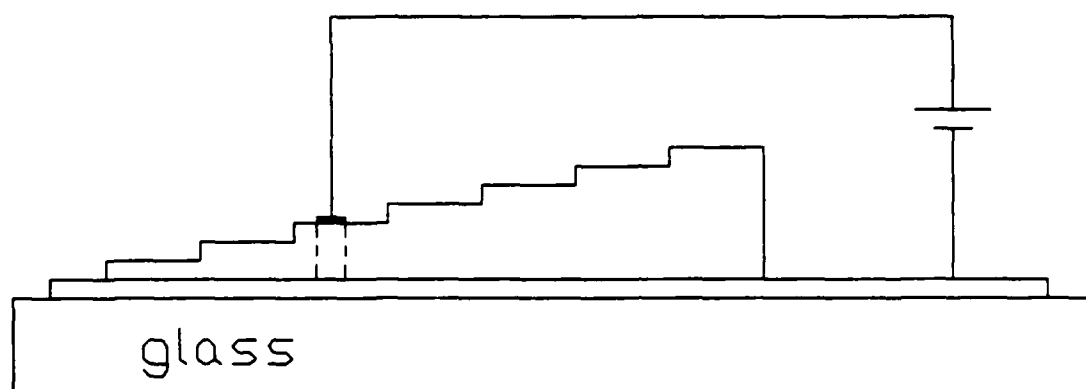
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DC device



Al	1000	Å
TCO	3000	Å
ZnS:Mn	5000	Å

Fig. 1 MIM device structure



Stepped sample  
Prepared in single pump-down

**Fig. 2 Stepped device structure**



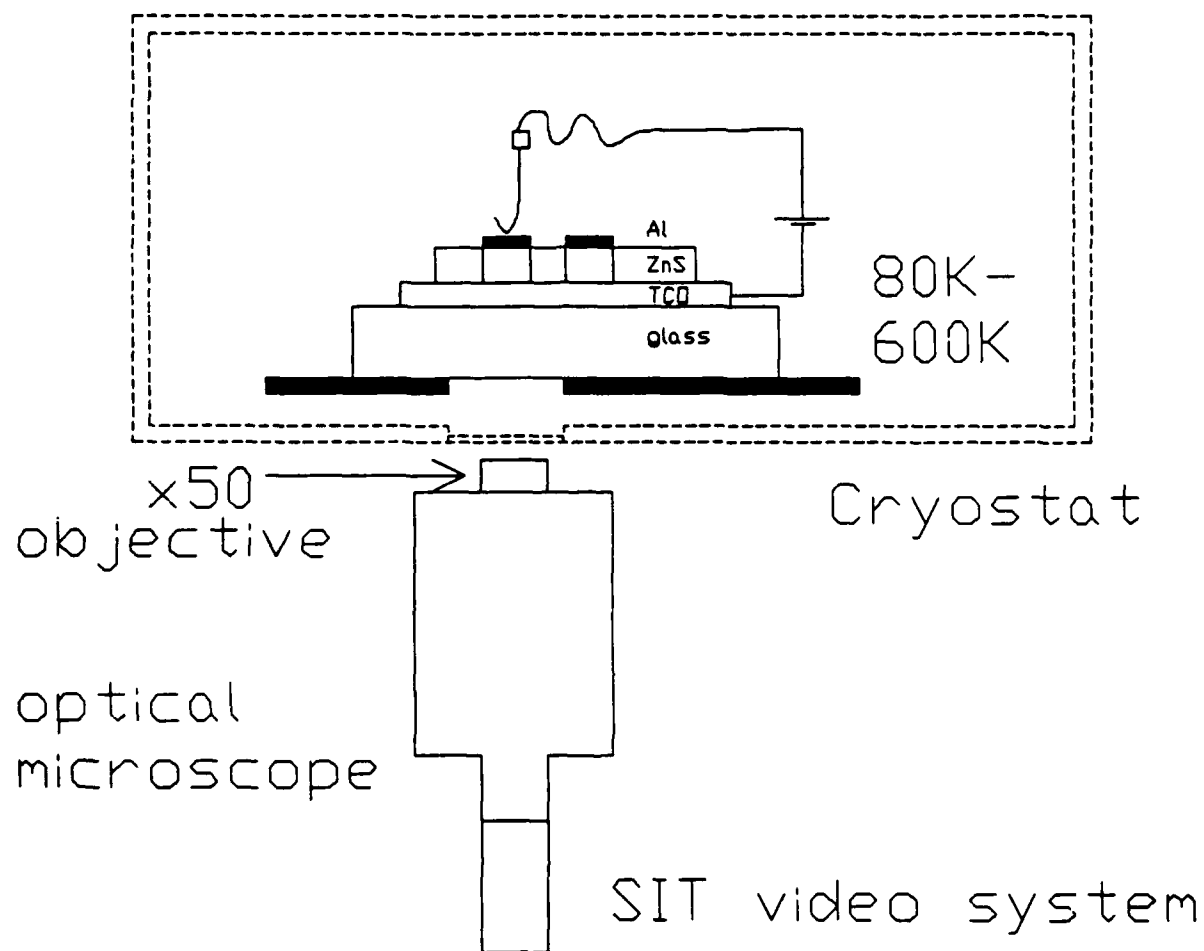


Fig. 3 Cryostat design

Constant voltage or  
constant current supplies

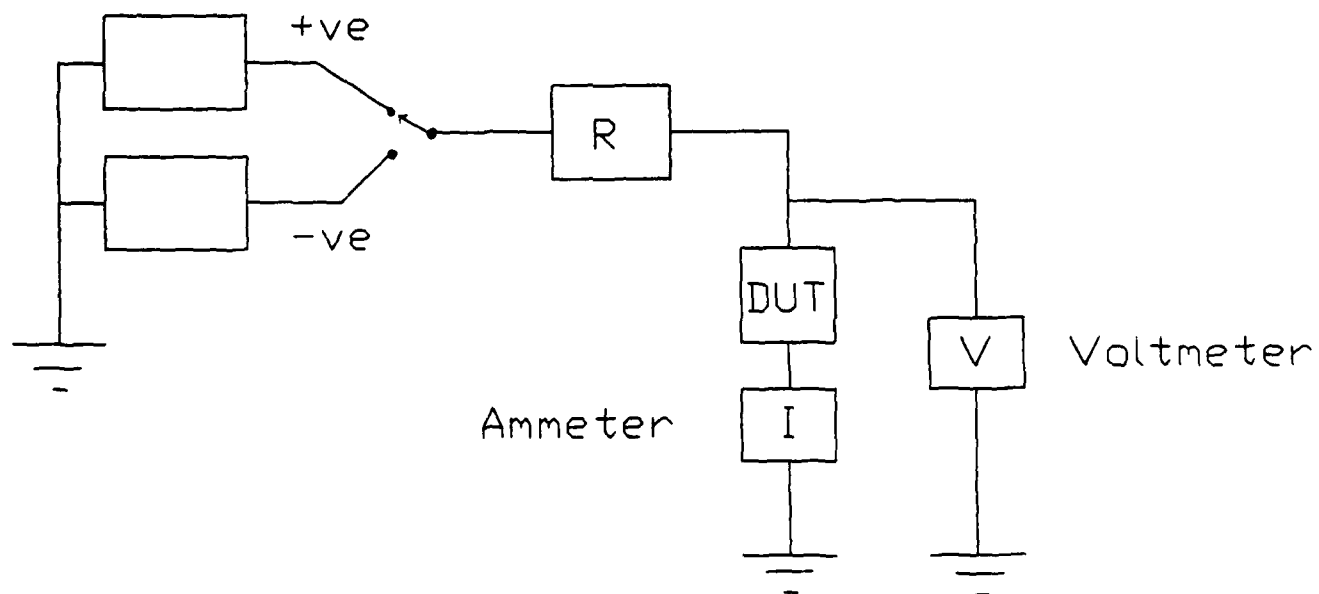
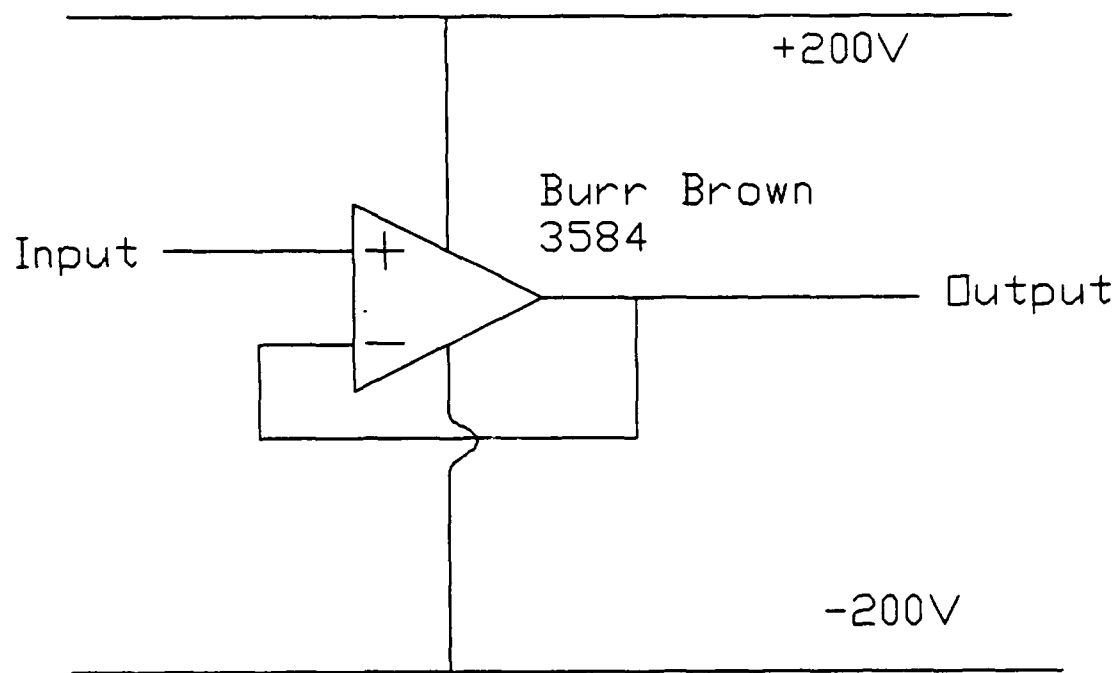
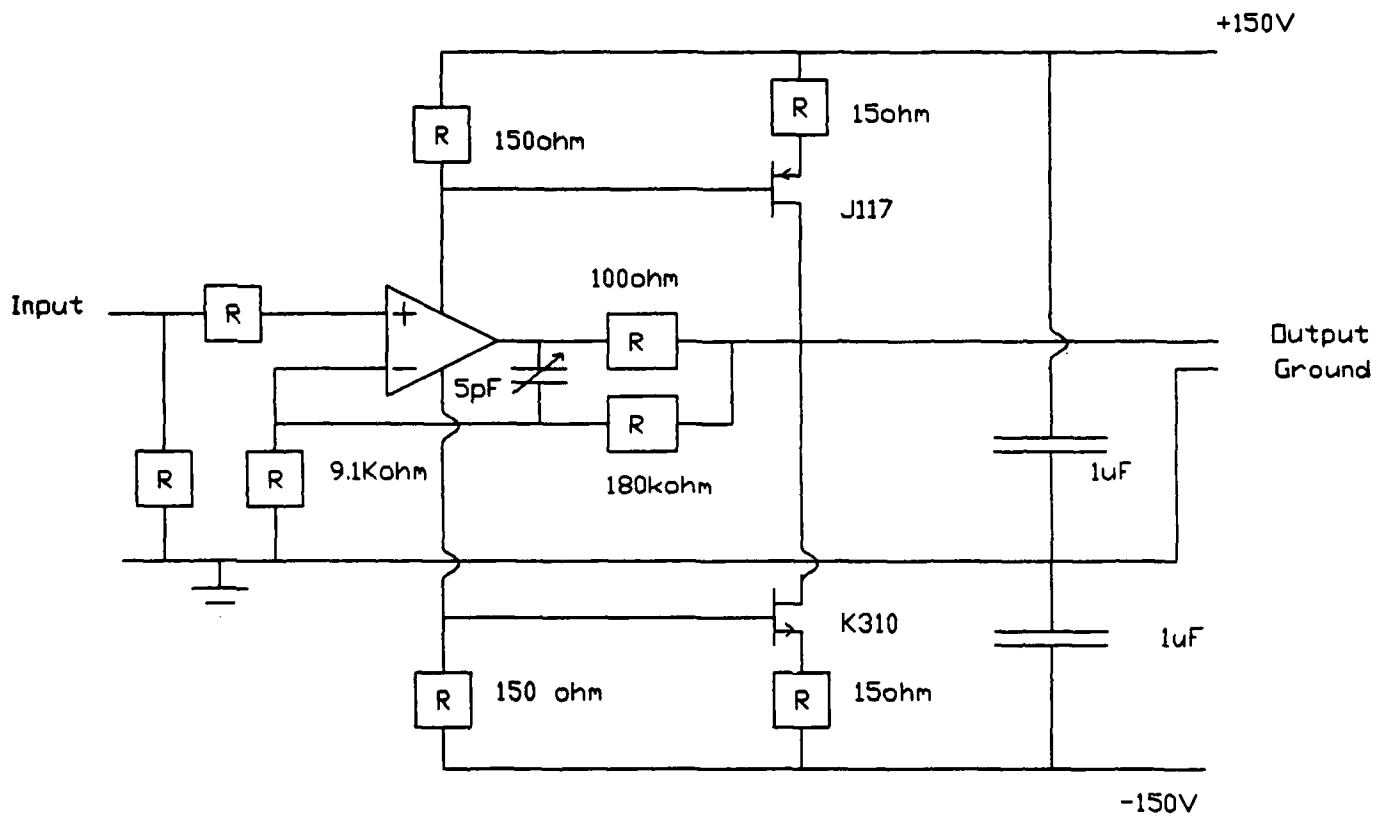


Fig. 4 Switching circuit



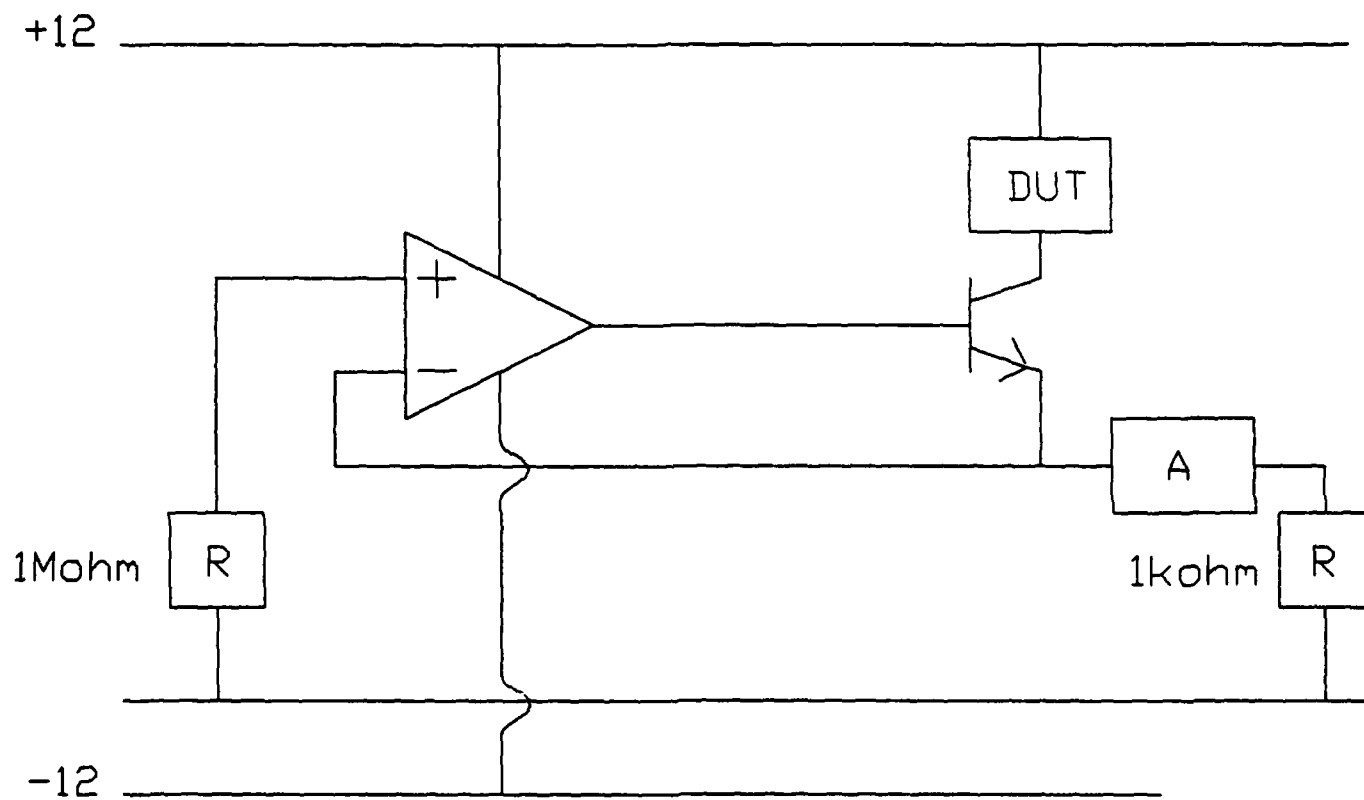
High voltage buffer amplifier

**Fig. 5 High voltage buffer amplifier**



High voltage pulse amplifier

Fig. 6 High voltage pulse amplifier



Constant current supply

Fig. 7 Constant current supply

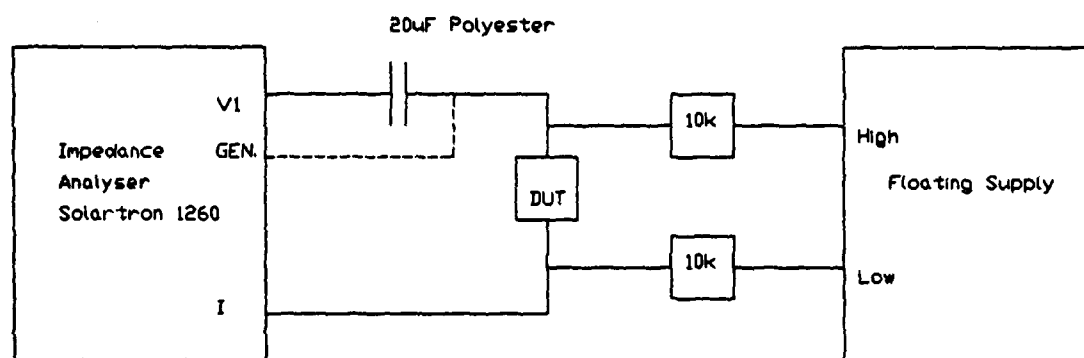
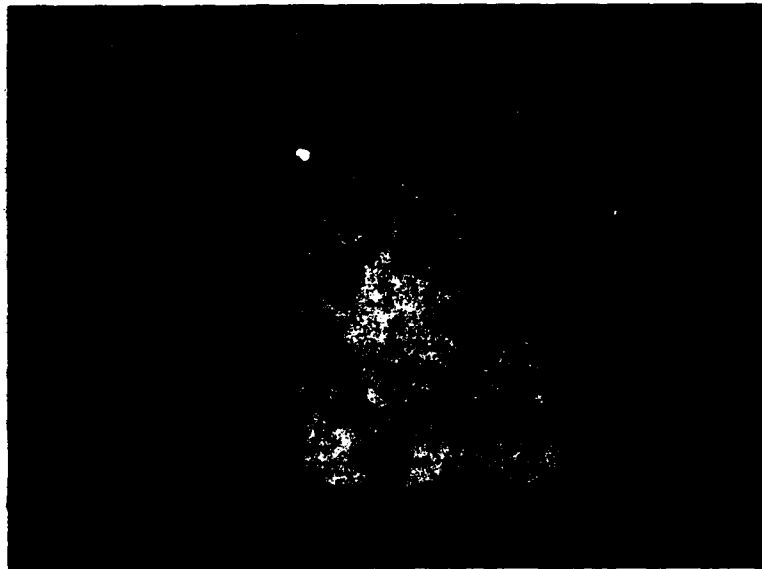


Fig. 8 Impedance analyser circuit



50  $\mu\text{m}$

Fig. 9 Example of interfacial contamination effects

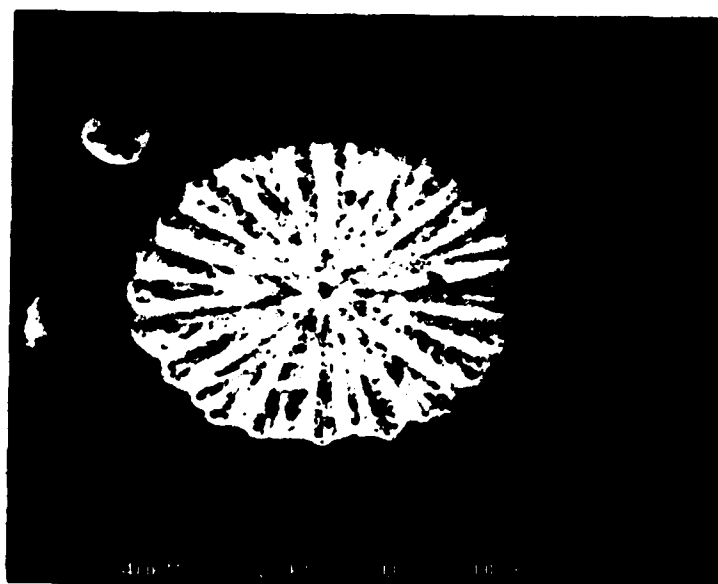
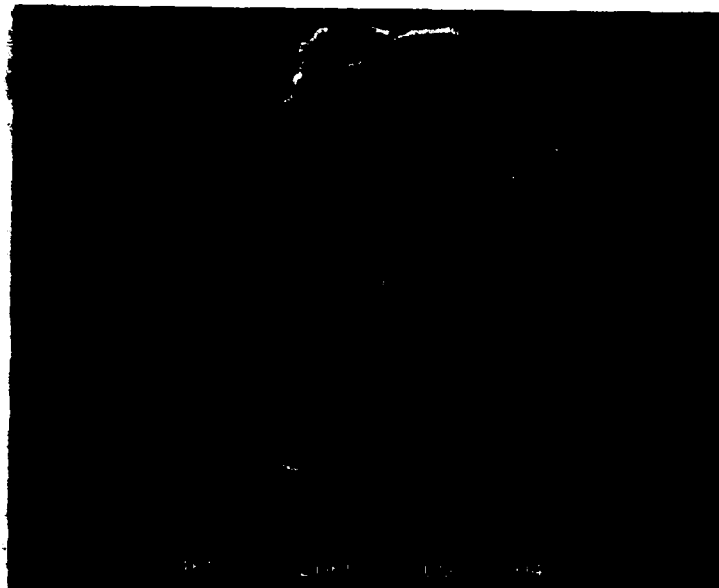


Fig. 10 Examples of self-healing breakdown craters





Fig. 11 Examples of kinetic electroluminescence; non-uniform current flow

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# REPORT DOCUMENTATION PAGE

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Originators Reference/Report No. MEMO 4401		Month JULY	Year 1990
Originators Name and Location RSRE, St Andrews Road Malvern, Worcs WR14 3PS			
Monitoring Agency Name and Location			
Title  TECHNIQUES FOR INVESTIGATING THIN-FILM INSULATORS UNDER HIGH ELECTRIC FIELDS			
Report Security Classification UNCLASSIFIED		Title Classification (U, R, C or S) U	
Foreign Language Title (in the case of translations)			
Conference Details			
Agency Reference		Contract Number and Period	
Project Number		Other References	
Authors BEALE, M I J			Pagination and Ref 29
Abstract  This memorandum describes techniques developed to investigate thin-film metal-insulator-metal devices operating under high electric fields and is intended as a practical guide to the experimentalist. Typically, the insulator will be a wide band-gap, optically active material with device applications including displays and electro-optic components. This memorandum contains a battery of approaches, some novel, some not, which provide a strategy for the acquisition of reliable data on this type of sample. Emphasis is placed on techniques to cope with the common problems of non-uniform conduction and of dielectric breakdown. Reference is made to the behaviour of the ZnS:Mn electroluminescent system to illustrate the techniques.			
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Descriptors			
Distribution Statement (Enter any limitations on the distribution of the document)  UNLIMITED			
880/48			